FINAL TECHNICAL REPORT

I. Objective of the Program

The objective of the research effort was to establish a fundamental understanding of high temperature fatigue in structural ceramics. Two additional closely related areas, environmental effect on fatigue and temperature dependence on strength and toughness, were also targeted where basic understanding needed to be acquired.

II. Research Accomplishments

We were successful in establishing a comprehensive framework as the basis for understanding fatigue and fracture over a broad range of temperatures, static/cyclic loading conditions, and environmental conditions. The premises of that framework were:

(i) Fatigue at low temperature is a process of mechanical balance in which resistance to crack advance is due to increased shielding whereas the impetus for crack advance is due to wear-caused degradation of crack wake shielding.

(ii) Fatigue at high temperature is a process of slow (creep) crack growth. Cyclic loading lowers the friction of grain boundary. The difference in crack growth rate in static and cyclic loadings can be rationalized by the different evolutions of crack-tip stress intensity factor under different grain boundary frictions.

(iii) Toughness in monolithic ceramics is due to grain pullout. At low temperature, the participating grains are ones that have broken grain boundaries due to prior crack deflection. At high temperature, most grains participate in pullout since the slip length of grain boundary sliding is long enough to trigger triple point separation.

(iv) Environmental effects in fatigue are similar to those in slow crack growth and are due to stress-assisted, thermally activated bond scission in the presence of an electron-donating environmental species.

The specific research accomplishments are as follows:

(a) **Fatigue Crack Growth of Silicon Nitride at 1400°C—A Novel Fatigue Induced Crack-Tip Bridging Phenomenon**: A high strength Si_3N_4 with elongated β-Si_3N_4 and equiaxed α'-sialon was tested in cyclic and static fatigue at 1400°C. At low stress intensity factors and high frequencies, the pull-out process of the elongated grains was enhanced which suppressed the crack growth. This provided a possible explanation for the increased lifetime under cyclic loading conditions reported for ceramics by several investigators. While crack-healing by high temperature annealing was found to greatly reduce the subsequent static fatigue crack growth rate, it had only a modest effect on cyclic fatigue and none at high frequencies.

(b) **Mechanical and Environmental Factors in the Cyclic and Static Fatigue of Si_3N_4**: The effects of environment on cyclic and static fatigue behavior were investigated with hot pressed silicon nitride materials. Tests were conducted at ambient temperature on standard compact tension specimens, and a DC electric potential technique was used to monitor crack lengths in-situ. The results indicate that the environmental sensitivity of our materials under both cyclic and static loading mirrored that of durable glasses in static fatigue. The materials were most sensitive to water in the environment, while changes in pH had no significant effect in the range tested. In addition, NH_3 was much less reactive with our materials than with vitreous SiO_2. In some cases, the intergranular glass appeared to be the site of environmental interaction. Evidence was also found that cyclic fatigue is not simply a manifestation of static fatigue. Cyclic fatigue was seen to occur in the absence of measurable static fatigue, and the data indicate that the mechanism of cyclic fatigue involves damage to the crack wake shielding zone.

(c) **Cyclic Fatigue in Ceramics: A Balance between Crack Shielding Accumulation and Degradation**: Cyclic fatigue growth rates in R-curve ceramics have been observed to depend very strongly on the maximum applied stress intensity, K_{max}, and only weakly on the stress intensity range, ΔK. This behavior is rationalized through measurement of crack wake shielding characteristics as a function of these fatigue parameters in a gas-pressure sintered silicon nitride. In particular, evidence for a mechanical equilibrium between shielding accumulation by crack growth and shielding degradation by frictional wear of sliding interfaces was found for steady state cyclic fatigue. This equilibrium gives rise to a rate law for cyclic fatigue. The data suggest that the accumulation process is the origin of the strong K_{max} dependence, and that the degradation process is the origin of the weak ΔK dependence. These features are shown to be related to the "cyclic" R-curve and to the cyclic crack opening displacement, respectively.

(d) **Effects of Temperature, Rate and Cyclic Loading on the Strength and Toughness of Monolithic Ceramics**: The concept of grain bridging and pullout was applied to monolithic ceramics to understand the effects of temperature, displacement rate, and load cycling on crack wake shielding. At low temperature, the pullout of completely debonded grains accounts for all the toughening. The importance of this process diminishes with temperature. This is because of the more uniform stress distribution along the crack plane and the softening of grain boundary glassy phases, both of which tend to reduce the incidence of complete grain boundary decohesion. At sufficiently high temperature, however, the softening of grain boundary phases may allow the sliding zone to extend to the grain's end, increasing the incidence of intergranular fracture. This "high temperature" pullout triggers a sudden increase in toughness. Our pullout model successfully explains the high temperature peak and the dependence of peak position on displacement rate in fracture toughness and strength observations for some monolithic and whisker-reinforced ceramics. Degradation of interfacial friction, as by cyclic loading, is seen to decrease the frictional work for low temperature pullout but increases the frictional work for high temperature pullout. Thus, this model also provides a rationale for the opposite effect of stress cycling on crack resistance at low and high temperatures reported recently for ceramics.

(e) **High Temperature Crack Growth in Silicon Nitride under Static and Cyclic Loading: Short-Crack Behavior and Brittle/Ductile Transition**: Crack propagation in Si_3N_4 at elevated temperatures was investigated using controlled surface flaws. Crack growth was generally slower under cyclic than static loading conditions. Concomitantly, there was a tendency for crack growth rate to initially decelerate despite an increasing driving force, exhibiting the so-called short-crack behavior. This tendency became more pronounced at higher temperatures, lower stress intensity factors, and larger cyclic stress variations. A corresponding transition in the crack profile, from a sharp one to a blunt one, was observed. These phenomena are attributed to enhanced crack-wake shielding due to grain pullout and the transition is believed to occur when the slip length of the grain boundary reaches the average half-length of the grain. The role of rising temperature and stress cycling then lies in the reduction of grain friction that in turn triggers more triple point separation and allows more grains to pull out. This picture is supported by fracture mechanics estimation of crack growth rate, crack opening displacement, and the characteristic length of the R-curve that constitute the short-crack behavior and brittle/ductile transition.

III. Publications

1. S.Y. Liu, I-W. Chen and T-Y. Tien, "Fatigue Crack Growth of Silicon Ntiride at 1400°C—A Novel Fatigue Induced Crack-Tip Bridging Phenomenon," *J. Amer. Ceram. Soc.*, **77** [1] 137-42 (1994).

2. D.S. Jacobs and I-W. Chen, "Mechanical and Environmental Factors in the Cyclic and Static Fatigue of Si_3N_4," *J. Amer. Ceram. Soc.,* **77** [5] 1153-61 (1994).

3. D. Jacobs and I-W. Chen, "Cyclic Fatigue in Ceramics - A Balance Between Crack Shielding Accumulation and Degradation," accepted for publication in *J. Amer. Ceram. Soc.* (1994).

4. I-W. Chen, S.Y. Liu, and D. Jacobs, "Temperature Dependence of Strength and Toughness of Monolithic Ceramics," in press, *Acta Metall. et Mater.* (1994).

5. S.Y. Liu and I-W. Chen, "High Temperature Crack Growth in Silicon Nitride under Static and Cyclic Loading: Short-Crack Behavior and Brittle/Ductile Transition," submitted to *Acta Metall. et Mater.,* 1995.

IV. Personnel

The program supported the following personnel:

I-Wei Chen, Principal; Investigator

Linda Malczewski, M.S. degree granted, September, 1991
Thesis entitled: *Uniaxial Tension-Compression Cyclic Fatigue of Alumina*

David Jacobs, Ph.D. degree granted September, 1994
Thesis entitled: *Mechanical and Environmental Factors in the Cyclic and Static Fatigue of* Si_3N_4

Xin Wu, Postdoc 1991-1992

Shyh-Yu Liu, Postdoc 1992-1994

V. Other Technical Presentations

A. Invited Presentations

1. "Constitutive Equations of Cyclic Fatigue of Ceramics," at the Engineering Foundation Conference on Fatigue of Advanced Materials, Santa Barbara, CA, January, 1991.

2. "Microstructural Design of Superplastic Ceramics," at the International Conference on Superplasticity of Advanced Materials, Osaka, Japan, June, 1991.

3. "Deformation Processing of Ceramics," at Purdue University, West Lafayette, IN, November, 1991.

4. "Tailoring of Grain Boundary and Bulk Properties by Grain Boundary Alloying," at Japan Fine Ceramic Center Workshop on Materials Processing and Design Through Better Control of Grain Boundaries," Nagoya, Japan, March, 1992.

5. "Fatigue of Tough Ceramics," in 15th Michigan Ceramic Materials Conference on Tough Ceramics, Ann Arbor, MI, March, 1992.

6. "Superplastic Forming of Ceramics," at the Winter Meeting of the American Society of Mechanical Engineers, Anaheim, CA, November, 1992.

7. "Superplastic SiAlONs," at the Materials Research Society Fall Meeting, Boston, MA, November-December, 1992.

8. "Cyclic Fatigue Degradation of Crack Tip Shielding in β-Silicon Nitride," at the Annual Meeting of the American Ceramic Society, Cincinnati, OH, April 1993.

9. "Sintering and Microstructural Development—Case Studies of Oxide and Nitride Ceramics," at the Annual Meeting of the American Ceramic Society, Cincinnati, OH, April 1993.

10. "Superplastic Structural Ceramics via a Transient Phase Approach," at the Symposium E (Superplasticity) 3rd IUMRS International Conference on Advanced Materials. Tokyo, Japan, August, 1993.

11. "Deformation of Fine-Grained SiAlON," at the Workshop on Tailoring of High-Temperature Properties of Si_3N_4-Ceramics. Schlossringberg, Germany, October, 1993.

B. Other Presentations

1. "Shear-Thickening Creep in Glass Containing Silicon Nitrides," S-L. Hwang* and I-W. Chen, at the 93rd Annual Meeting of The American Ceramic Society, Cincinnati, OH, April-May, 1991.

2. "Transient-Liquid Aided Superplastic Forming of Silicon Nitrides," S-L. Hwang* and I-W. Chen, at the 93rd Annual Meeting of The American Ceramic Society, Cincinnati, OH, April-May, 1991.

3. "Formability of Superplastic Si_3N_4 in Uniaxial and Biaxial Deformation," X. Wu* and I-W. Chen, at the 93rd Annual Meeting of The American Ceramic Society, Cincinnati, OH, April-May, 1991.

4. "Uniaxial Tension-Compression Cyclic Fatigue of Alumina," L.J. Malczewski* and I-W. Chen, at the 93rd Annual Meeting of The American Ceramic Society, Cincinnati, OH, April-May, 1991.

5. "Environmental and Mechanical Contributions to Cyclic and Static Fatigue in Si_3N_4 and Al_2O_3," D. Jacobs* and I-W. Chen, at the 94th Annual Meeting of The American Ceramic Society, Minneapolis, MN, April, 1992.

6. "Crack Propagation of a High Strength Si_3N_4 at 1400°C Under Creep and Fatigue Conditions," X. Wu,* T-S. Sheu, T.Y. Tien and I-W. Chen, at the 94th Annual Meeting of The American Ceramic Society, Minneapolis, MN, April, 1992.

7. "Fatigue of Structural Ceramics," S.Y. Liu,* D. Jacobs and I-W. Chen at the TMS Annual Meeting, Denver, CO, February, 1993.

8. "High Temperature Fatigue of Structural Ceramics," S-Y. Liu,* I-W. Chen, and J. Calomeni-Michels at the 95th Annual Meeting of The American Ceramic Society, Cincinnati, OH, April, 1993.

9. "High Temperature Mechanical Properties of Pressureless-Sintered Si_3N_4 Composites," S.Y. Liu,* Z.K. Huang, and I-W. Chen at the 96th Annual Meeting of the National Institute of Ceramic Engineers, Indianapolis, IN, April, 1994.

10. "High Temperature Fatigue Properties of a SiC Whisker Reinforced Li-Aluminosilicate," J. Calomeni-Michels,* S.Y. Liu, and I-W. Chen at the 96th Annual Meeting of The American Ceramic Society, Indianapolis, IN, April, 1994.

11. "Fatigue of Si_3N_4 at Elevated Temperatures: Short-Crack Behavior and Ductile-Brittle Transition," S.Y. Liu* and I-W. Chen at the 96th Annual Meeting of The American Ceramic Society, Indianapolis, IN, April, 1994.

12. "Cyclic Fatigue in Al_2O_3: A Balance between Degradation and Accumulation of Crack Wake Shielding," D.S. Jacobs* and I-W. Chen at the 96th Annual Meeting of The American Ceramic Society, Indianapolis, IN, April, 1994.

13. "Temperature Dependence of Strength and Toughness of Monolithic Ceramics," S.Y. Liu* and I-W. Chen at the 96th Annual Meeting of The American Ceramic Society, Indianapolis, IN, April, 1994.

14. "Phase Relationships in Part of Sm, Si, Al/N, O System and Their Implications for Sm-SiAlON Fabrication," Z.K. Huang* and I-W. Chen at the 96th Annual Meeting of The American Ceramic Society, Indianapolis, IN, April, 1994.

REPORT DOCUMENTATION PAGI

AFOSR-TR-95 0626

188

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
March 9, 1995

3. Final Technical Report 1/7/91 - 1995

4. TITLE AND SUBTITLE
High Temperature Fatigue of Structural Ceramics

5. FUNDING NUMBERS
AFOSR-91-0094

6. AUTHOR(S)
I-Wei Chen

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
University of Michigan
Department of Materials Science & Engineering
2300 Hayward St.
Ann Arbor, MI 48109-2136

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Office of Scientific Research
Ceramics and Non-Metallic Structural Materials Program
110 Duncan Avenue, Suite B115
Bolling Air Force Base, DC 20332-6448

NA

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
AFOSR-91-0094

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unrestricted

Approved for public release, distribution unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

Fatigue research on monolithic ceramics has been conducted using Si3N4 and Al2O3. At low temperature, fatigue is due to wear-causeddegradation of grain sliding friction that decreases crack wake shielding. At the steady state, this degradation is balanced by increased crack tip shielding as crack advances. At high temperature, fatigue is due to slow crack growth driven by the crack tip stress intensity factor. Fatigue retardation and short crack behavior arises in the latter case because of more active grain boundary sliding and triple point cracking caused by degraded sliding resistance under cyclic loading conditions. The overall dependence of crack growth rate on temperature, strain rate, cyclic load ration, and environmental chemistry of structural ceramics has been delineated and interpreted in fundamental terms based on the above micromechanisms.

14. SUBJECT TERMS
Fatigue, Ceramics, Si3N4, Al2O3, High temperature, Environment, Toughness, Strength

15. NUMBER OF PAGES
6

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT

18. SECURITY CLASSIFICATION OF THIS PAGE

19. SECURITY CLASSIFICATION OF ABSTRACT

20. LIMITATION OF ABSTRACT